# Analysis of Damage Behavior Based on EBSD Method and the Law of Fracture Life under Creep-Fatigue Conditions for Polycrystalline Nickel-base Superalloys



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# 論 文 内 容 要 旨

Developments in gas turbine technology in recent years are remarkable. In Japan, the introduction of combined cycle power plants with LNG fuel 1100°C-class gas turbines in 1984, was followed by the increasing installation of 1300°C-class combined cycle power plants in the 1990s, and currently the 1500°C-class gas turbines have started commercial operation. Currently, fundamental development of the 1700°C-class gas turbine has been facilitated as a national project. The attractiveness of combustion turbines, as viable power-generation options in today's context, can be attributed to a number of factors, but mainly they are relatively inexpensive and quick to install. This gives the utility companies enormous flexibility in the face of uncertainties concerning other power source development. Combustion turbines constitute the main part of the total power-generation capacity of Japan's electric utility industry.

As the combined power plants are advanced, life assessment techniques for combustion turbine are becoming more important. There is an urgent need for techniques to assess the condition of components in turbines currently in service. Also, there has been a growing need to assess accurately the remaining lives of aging components. The need for life-assessment technology is driven by many economic factors. Several turbine blades currently in operation have been in service for more than 50,000 hours and are likely to perform reliably even after exceeding their original design life. A properly managed life-assessment and refurbishment program may extend the life of these blades. Turbine blades and disks are highly stressed components whose failure can lead to more consequential damage. Hence, basically, the failure criteria of blades and disks concern the problem of crack-initiation. Because of the enhancement of inner cooling systems with elevating gas turbine inlet temperatures, localized thermal stress and its damage progression become important issues. An important fact to remember is that neither the stress nor the temperatures in blades or disks are uniform. Both of these parameters vary along and across the components, which result in gradients. Sometimes these gradients are confined to local regions around cooling holes, local hot spots or other design features, which promotes localization of creep damage. Operating practices also vary widely among utilities or individual plants from base load duty (time dependent damage mechanisms) to peaking duty (cycle dependent damage mechanisms) depending on the energy demands at that time. In such cases, utilities need the technology to assess the effects of time-dependent and cycle dependent damage progression on a unit-specific basis. However, it is difficult to comprehend the characteristics of the load frequency of the fracture life under creep-fatigue conditions, since the diverse characteristics include such factors as stress, stress holding time, material properties and temperatures. In many cases, this relationship is under complex interactive conditions due to cycle- and time-dependent mechanisms.

Nickel-base superalloys that are precipitation strengthened by the γ' phase in grains have a pronounced brittle property even at high temperatures, therefore creep degradations such as deformation of components, initiation of microscopic creep voids and cracks only appear at the end of the available creep fracture life. Therefore, in order to assess accurately the integrity of a target component, it is important to find indications of creep degradation or evidence of some kind of damage. The essence of damage progression is considered to be the growth of irreversible alteration such as inelastic strain. It is also known that inelastic deformation throughout crystal grains is not homogeneous.

The aim of the current work is to obtain comprehensive knowledge of microscopic damage progression behavior before a crack initiation, and to derive the laws of fracture life under creep-fatigue conditions for polycrystalline nickel-base superalloys, which will contribute to assess the integrity of turbine components and to conduct accurate life assessments of those hot-gas-path components. Particular attention was paid to the laws of fracture life under creep-fatigue conditions, the quantitative evaluation of creep and/or fatigue damage and the estimation method of fracture modes under various applied stress conditions. We revealed that these techniques are valid for actual turbine materials through experiments and theoretical analyses.

In Chapter 2, to clarify the experimental background of characteristics of fracture life of the investment-cast polycrystalline nickel-base superalloy IN100 and IN738LC (typical gas turbine blade's materials), in-situ observational tests under creep-fatigue conditions were carried out, and the effects of both the cycle- and the time-dependent mechanisms on the fracture life were investigated. It has been found that creep ductility, stress holding time ($t_H$), and temperature (T) were unified as a promoting factor of the time-dependent mechanism: P, which was then combined with the relationship between the inverse value of fracture life and applied load frequency. Then a three-dimensional curved surface representation of fracture life under arbitrary creep-fatigue conditions has been proposed theoretically according to the non-equilibrium science. Finally, the multiple effects of creep and fatigue on the crack growth behavior and fracture life were clarified. As the load frequency decrease or stress holding times and temperature increases, time dependent mechanism begins to play a role in the manner of creep-fatigue interaction. In this region, the characteristics of the relative notch opening displacement (RNOD) and crack growth behavior change from a cycle-dependent mechanism caused by fatigue to a time-dependent mechanism caused by creep through an unstable equilibrium transition region. A predictive law of the fracture life under creep-fatigue condition was derived as a three-dimensional curved surface representation of the load frequency characteristic for IN100 and IN738LC. Comparison of the theoretical results with experimental results shows that the transition behaviors of load frequency on fracture life were characterized with the parameter of P which shows that the degree of the involvement of the time-dependent mechanism increases with an increase in temperature or stress holding time. Regarding IN100, the transition behaviors of load frequency on fracture life under temperatures of 800°C to 900°C were characterized as a change of P. In addition, under creep-fatigue conditions, three-dimensional curved surface representations support the notion that a creep crack growth mechanism appears at over 760°C. Also, regarding IN738LC, by deriving three-dimensional curved surface representation (Fig.1), it was shown that the theory also supports the notion that under creep-fatigue condition, a creep crack growth mechanism appears at over 678°C.

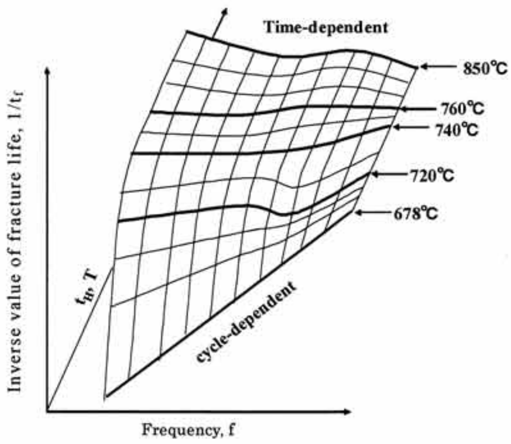

Figure 1 Three-dimensional curved surface representation of load frequency characteristic of IN738LC

In Chapter 3, in order to detect creep damage caused by vacancy diffusion before crack initiation stage, investigation into criteria of crack initiation, that is, creep deformation and local strain including the characteristics of the RNOD was carried out. Although those criteria of crack initiation against actual components cannot be measured, with the current advances in electron backscattered diffraction (EBSD), it has become possible to make unprecedented submicron resolved measurements of the local crystal structure distribution at component's thickness (millimeter) scale. By making full use of these advances, attempts have been made to measure inelastic strain as changes in crystallographic orientation. The effects of test conditions such as temperatures, strain rates and stress holding times, which make it difficult to achieve quantitative damage evaluation, are also investigated. Furthermore, the influence of the geometry of stress concentrated regions and creep-fatigue interactions on misorientation development has confirmed by observing many interrupted smooth and notched specimens. In the creep interruption tests, some specific creep specimens were made individually. In addition, in the case of the creep-EBSD repeat tests, a notched specimen was alternately subjected to a repeated specific creep strain and EBSD observation. Finally, quantitative investigation to assess the remaining creep fracture life of the nickel-base superalloy was carried out. The proportional relationships between creep strain and misorientation of smooth samples under various temperatures and strain rates conditions were obtained in term of the grain reference orientation deviation (GROD) parameter. Although localization of misorientation along grain boundaries was observed, as long as the vacancy diffusion dominates the creep deformation, the macroscopic strain balances with the average misorientation of the cross section under high temperature conditions. Misorientation development of notched specimens was localized in the stress concentrated region and grain boundaries (Fig.2) depending on the initial stress concentration factor. However, analysis of the whole area of notched specimens allows the uniform evaluation of microscopic creep damage before the appearance of obvious defects such as voids or cracks without being subjected to the geometrical influence of stress concentrated region as shown in Fig.3. In addition, the average misorientation within grains evaluated by the parameter of GROD increases linearly up to the initiation of cracks with the increase of creep strains regardless of the testing temperatures and strain rates or creep-fatigue interaction except pure fatigue condition which shows obvious crack growth behavior. It is concluded that the misorientation analysis of damaged materials based on the EBSD method allows the quantitative estimation of creep strain and the assessment of remaining creep fracture life.

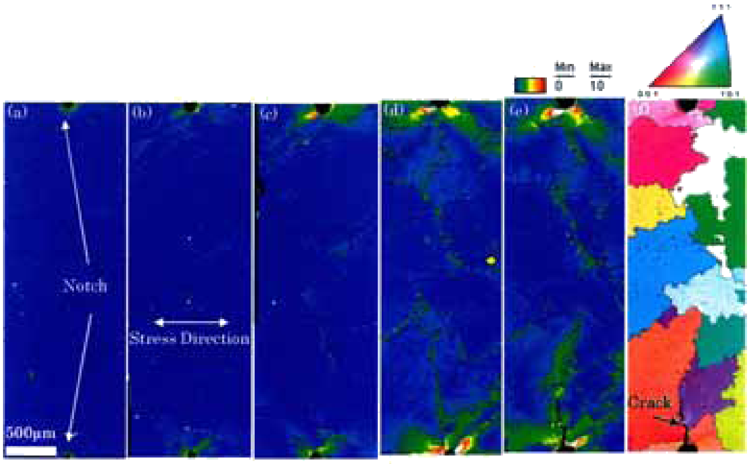

Figure 2 GROD maps of each creep life fraction on a creep-EBSD repeat test at 830°C×294MPa ($t_f$≒110h), (a) $t/t_f$=0.0, (b) $t/t_f$=0.22, (c) $t/t_f$=0.45, (d) $t/t_f$=0.88, (e) $t/t_f$=0.99, and (f) IPF map at $t/t_f$=0.99.

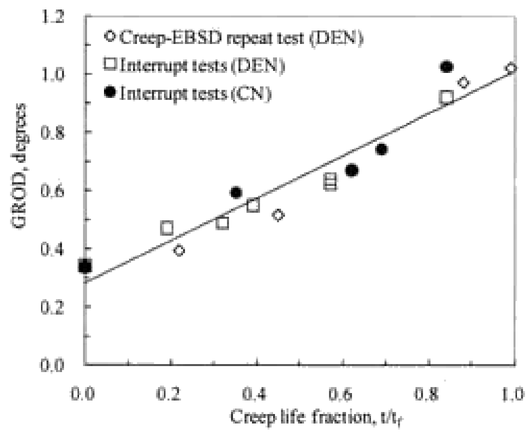

Figure 3 Creep life dependence of GROD (analysis area average) using double-edge-notched (DEN) and center-notched (CN) specimens.

In Chapter 4, in order to complement the experiment conducted under constrained conditions, we have tried to develop the predictive method of misorientation development by numerical analysis using finite element analysis (FEA). To clarify the initiation and growth tendencies of the microscopic inhomogeneous deformation, the FEA has taken account of the effects of individual grains. In addition, the physical meaning of misorientation parameters has been clarified. In the numerical analysis, the difference of properties depending on each grain is given by the elastic-plastic anisotropy of individual grains instead of orientation inhomogeneous depending on crystal orientation. Virtual misorientation is initiated not only around the notch tip but also at the boundaries between elastic elements and creep elements near grain boundaries. This result is in good agreement with that of actual creep damage progression behavior as shown in Chapter 3. In addition, by the proposed simulation method, the influence of grain structure or notch shape on misorientation development can be evaluated in detail. The GROD is qualitatively in good agreement with that of inelastic strain and it is physically corresponding to the creep damage as shown in Fig.4 (a) and (b). The large value of GROD is expected to correspond to the region where macroscopic main crack will initiate and grow. Also, the theoretical kernel average misorientation ($KAM_{th}$) by numerical analysis has large value along the boundaries between elastic element and inelastic element as shown in Fig.4 (c) and (d). Therefore, the large value of KAM in experiment is considered to show the region where the driving force of creep damage is significant. In addition, the large value of KAM is expected to correspond to the region where microscopic damage such as creep voids and micro cracks will occur.

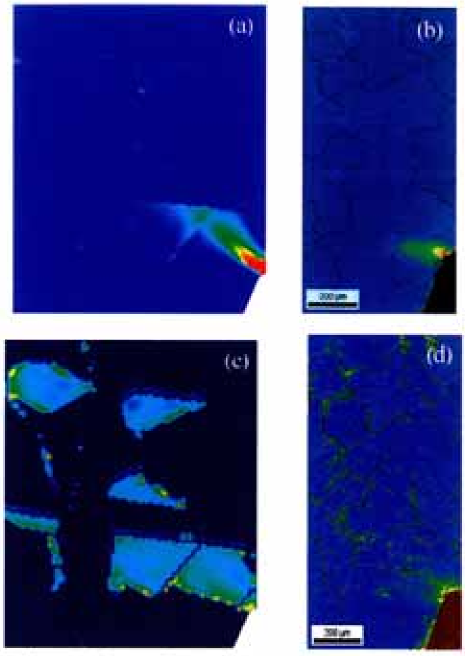

Figure 4 Comparison of the analytical results and experimental characteristics of creep damage, (a) Inelastic strain, (b) GROD map, (c) $KAM_{th}$ map by numerical analysis and (d) KAM map by EBSD.

In Chapter 5, in order to make clear the cause of the fracture of a polycrystalline nickel-base superalloy, whose fractography analysis was not possible, investigations into the cross sectional analyses of various mechanical fractured specimens have been carried out. In addition, investigation into the crack growth mechanisms along the grain boundary has been carried out. Tensile fracture samples of both IN738LC and IN706 which is the typical gas turbine disk's materials made of polycrystalline forged nickel-base superalloy were deformed over the whole zone of the parallel portion of the specimen and showed quite large plastic deformation. The plastic slip mechanism at room temperature or significant vacancy diffusion along grain boundaries under high temperatures was found to dominate the tensile deformation. The impact fracture samples of both IN738LC and IN706 showed quite large plastic strain only at the vicinity of the fracture surface. This constraint of damage propagation is considered to be caused by dynamic loading. Creep damage affected a large area of the specimens but was localized near grain boundaries in IN738LC, although the IN706 shows the grain boundary embrittlement with no change of crystal orientation. The misorientation profile analysis of the cross section of fracture samples using the EBSD misorientation analysis allows the qualitative estimation of the fracture mode as shown in Fig.5 and Fig.6. It is expected to clarify the damage mechanism by advanced crystallographic misorientation analysis using the EBSD method.

In Chapter 6, concluding remarks concerning the current work are given.

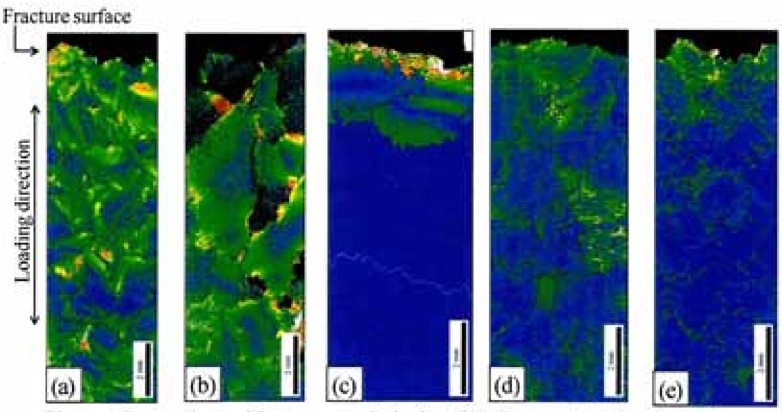

Figure 5 Comparison of fracture morphologies (GROD maps), (a) Tensile: RT , (b) Tensile: 800°C, (c) Impact: RT, (d) Creep: 760°C and (e) Creep: 980°C.

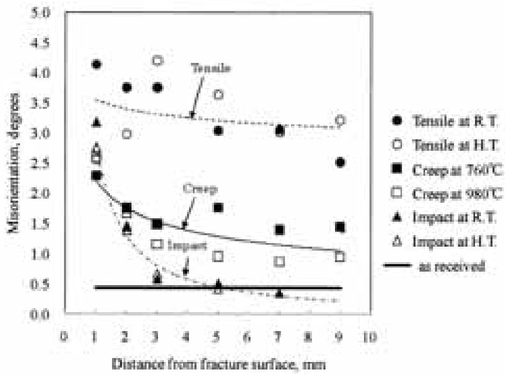

Figure 6 GROD misorientation distributions with distance from the fracture surface of IN738LC.

# 論文審査結果の要旨

Ｎｉ基超合金は，高効率発電におけるガスタービン翼材など高温構造用材として用いられている。しかし，高温脆性材料であるため巨視的変形やき裂成長段階は，すでに最終破壊状態になるため早期の微視損傷状態での寿命予測が必要になる。著者は，実機で問題となる高温クリープ・疲労相互作用条件下での破壊寿命を予測する独自の方法を系統的な実験と非平衡科学を基にした理論により提案し，さらにEBSD法により微視損傷を検出できることを示し，これを，破壊機構と関連させている。本論文はこれらの成果をまとめたものであり，全編6章からなる。

第1章は緒論であり，本研究の背景および目的と意義について述べている。

第2章では，多様に変化するクリープ・疲労相互作用条件下での破壊寿命の繰返し速度特性を時間依存促進パラメータ，Pを新たに導入して，破壊寿命，繰返し速度およびPを3次元空間に表示し，この曲面を表す理論式を非平衡科学の理論を用いて導出している。これにより，クリープ・疲労相互作用条件下での破壊寿命を理論的に予測できるばかりではなく，実機最適運転温度に関わるクリープが発現しにくい限界温度を理論的に予測できることを示している。3次元表示法と非平衡科学を基にする理論式は，独創的な提案であり，工学的に重要な成果である。

第3章では，クリープ・疲労条件下での系統的な途中止め実験を行い，各変形段階における結晶方位差指標（GROD，KAM）の分布をEBSD法により求めている。これらのデータを解析して，GROD値の時系列変化特性がクリープ・疲労条件下での変形特性と対応することを示している。本結果は，EBSD法により解析されるGROD値がクリープ･疲労における微視損傷量と対応することを示すものであり，微視損傷観察と評価の可能性を与える重要な成果である。

第4章では，EBSD法により測定される応力集中部近傍でのKAMおよびGROD値の局所分布と蓄積損傷量に関わる力学量との対応について試作改良した結晶方位の影響を考慮した弾塑性クリープ有限要素解析プログムを用いて解析している。本解析により高KAM値領域は，クリープ損傷領域を表し，高GROD値領域は高クリープひずみ領域（損傷進行度）を表すことを明らかにしている。本結果はEBSD法による微視損傷計測法を提案するものであり，工学的に有用な成果である。

第5章は，弾塑性破壊，衝撃破壊，低サイクル疲労およびクリープ破壊において，EBSD法により計測されるGROD値の分布領域で特定される微視損傷形成領域について比較検討を行っている。本結果は微視損傷形態の相違から破壊に至った原因を明らかにすることを可能にするもので，破壊原因の解明に有用な成果である。

第6章は結論である。

以上要するに本論文は，　高温クリープ・疲労の非線形的相互作用効果を取り込んだ破壊寿命則を提案し，さらに，破壊に至るまでの微視損傷を計測する方法を提案したもので，高効率発電機器の安全性，信頼性の向上に有益なものであり，ナノメカニクスおよび機械工学の発展に寄与するところが少なくない。

よって，本論文は博士(工学)の学位論文として合格と認める。